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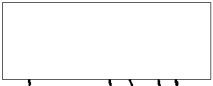
SUMMARY REPORT

ON

RESEARCH ORDER NO. 29

FINAL REPORT FILE

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Controlled Dehumidification
June 30, 1957

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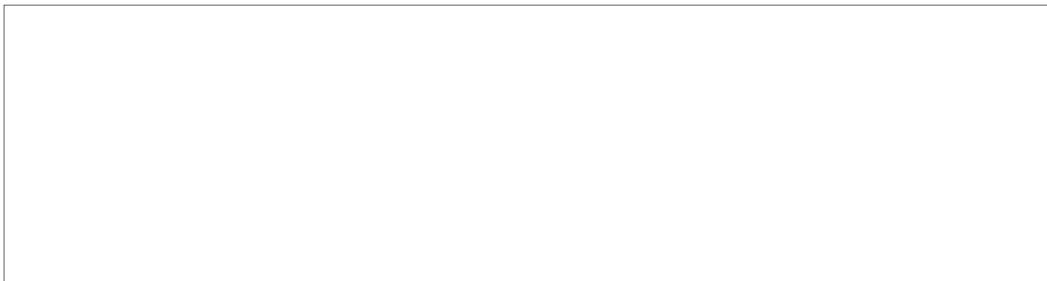
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SECRET**SUMMARY REPORT**

on

RESEARCH ORDER NO. 29

June 30, 1957

INTRODUCTION

In the Sponsor's operations, it is sometimes necessary to store small equipment and materials under adverse conditions for considerable periods of time. This storage is best effected in an atmosphere of low relative humidity within closed containers. Research Order No. 29 was set up to develop a desiccating capsule that would maintain a low relative-humidity atmosphere within a closed container, thus preventing the deterioration of equipment and materials during long-term closed storage.

This report summarizes the activity performed under Research Order No. 29, during the period from December 1, 1955, through June 30, 1957.

DETAILED SUMMARY

Silica gel was chosen as the desiccating agent for investigation. This material is chemically inert, relatively inexpensive, and commercially available in different types and sizes of packages. Of the various grades of silica gel, Protek-Sorb 121 was evaluated in this study, primarily because of its high capacity to adsorb water vapor in a closed system; this grade also removes vapors other than water vapor from an enclosed atmosphere, and satisfies Specification MIL-D-3464.

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In the performance of the research, four containers representing two types (with approximately 0.6 and 2.3-cubic-foot internal capacity) were packed with a variety of items, including pistols, binoculars, batteries, flashlights, electronic components, hand tools, metallic and nonmetallic materials, and others; silica gel was loaded into three of the containers. The packing was done under temperate and under simulated-tropical conditions, i. e., at room temperature and about 50 per cent relative humidity, and at 90 F and about 80 per cent relative humidity, respectively. The packed containers were then stored continuously for 8 hours of each day at about 120 F followed by 16 hours at about 40 F. Relative-humidity readings were taken at the end of each storage period at each temperature using humidity-sensing elements, which had been built into the containers, and a humidity indicator; the humidity-measuring equipment had been procured from the American Instrument Company, Incorporated, Silver Spring, Maryland.

In preliminary experiments, various amounts of silica gel were used. Ultimately, it appeared that about 300 grams of Protek-Sorb 121 per cubic foot of internal container capacity would provide the desired degree of control of the relative humidity inside the containers. Experiments were then conducted for a period of approximately six months using this "concentration" of silica gel. At the end of this cycled-storage period, all of the stored items were examined qualitatively, and some of the electronic components quantitatively.

The approximately 300 grams of silica gel per cubic foot maintained the relative humidity in the three containers below 35 per cent during the six-month period, as compared to 70 to 72 per cent in the container without silica gel. However, the relative-humidity data showed an

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apparent increase with increasing storage time. This apparent increase is attributed to three factors, only one of which pertains to the effectiveness of silica gel in this type of application under service conditions. This factor is the strong tendency of water vapor to migrate from a relatively humid atmosphere through a liquid-water-tight seal to a relatively dry atmosphere. On the basis of calculations, it is believed that the use of an additional 150 grams of Protek-Sorb 121 per cubic foot would maintain adequate control over the amount of water vapor involved as a result of this permeation tendency, for a period of approximately 24 months at a temperature of 120 F.

In general, most of the items packed within the four containers under the above-described conditions were in good condition at the end of the six-month cycled-storage period; the electronic components exhibited only normal variations due to storage. Some of the items, such as B and C batteries, and batteries stored within flashlight cases, showed serious physical and electrical deterioration, even when silica gel was used. Items such as pistols and steel-cased flashlight batteries stored separately (i. e., not within flashlight cases) were in good condition when silica gel was present; when silica gel was not used, the pistol was inoperative and the flashlight batteries were dead. None of the items appeared to be significantly damaged from storage in the relatively dry atmosphere resulting from the presence of silica gel; the Scotch cellophane tape and the medical adhesive tape showed decreased adhesion, but these materials were still quite usable.

Cycled-storage experiments were also conducted on about 0.5 cubic feet of each of four cushioning materials loaded in the 0.6-cubic-foot

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containers under simulated-tropical conditions (90 F and about 80 per cent relative humidity); approximately 300 grams of Protek-Sorb 121 per cubic foot of internal capacity was used in each container. During one month of cycled storage, the containers packed with Kimpak and Hair-lok showed 40 to 50 per cent relative humidity, and those packed with normal and non-water-absorbent grades of Tufflex yielded relative-humidity values above 70 per cent. It appears that the two grades of Tufflex would not serve as well as would Kimpak or Hair-lok, under severe conditions involving tropical loading and long-term closed storage.

Silica gel is commercially available in tough, resin-impregnated, water-vapor-permeable bags, or in water-vapor-permeable, perforated aluminum containers. After the Sponsor indicated that the use of a metal container to hold the silica gel would be advantageous, bags were eliminated from further consideration as containers for the silica gel. Protek-Sorb 121 is marketed in 24.8-gram quantities packed in aluminum containers of two shapes, circular and rectangular in cross section. For shipping and storage purposes, these packets are enclosed in friction-top metal cans that conform to the shape of the silica gel packets; plastic tape is wrapped around the edges of the outer cans.

A few experiments were performed to obtain some indication of the resistance of the commercial packaging arrangement to moisture permeation. Cans containing silica gel packets of both shapes were stored, with and without the sealing tape intact, under simulated-tropical conditions (90 F and about 80 per cent relative humidity) for one month. The results indicated that, from the viewpoint of water-vapor pickup during shelf storage, the circular-cross-sectioned metal container with the sealing tape intact represented the best of the packaging arrangements investigated.

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It appears that a "standard" silica gel package which the Sponsor could use effectively would consist of 80 to 100 grams of Protek-Sorb 121 packed in a circular-cross-sectioned, perforated metal container. For shipping and storage purposes, such packages would be enclosed in a circular-cross-sectioned, friction-top, metal can, with plastic sealing tape wrapped around the periphery.

On the basis of the data obtained in this research program, and of appropriate calculations, selected pertinent estimates have been made. It appears that adequate relative-humidity control at 40 to 120 F for about 24 months would be maintained in small and large containers (0.6 and 2.3-cubic-foot internal capacity, respectively), such as those used in the above-described experiments, by approximately 2 to 4 and 7 to 13 "standard" 80 to 100-gram packages, respectively. The approximate volumes that would be occupied by these numbers of "standard" packages would be 30 to 48 and 105 to 165 cubic inches, respectively; these would correspond to less than 5 per cent of the internal capacity of either of these storage containers.

To explore some of the potential logistics problems with regard to procurement of "standard" silica gel packages by the Sponsor, a manufacturer of silica gel was contacted. The organization indicated a definite interest in and willingness to produce silica gel packages that were different from their current production items.

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DISCUSSION OF THE PROBLEM

The main cause of deterioration of equipment and materials under the particular service conditions of storage is probably water vapor, either by itself or possibly in conjunction with other vapors that may be present

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in the enclosed atmosphere. The water vapor adsorbed on or absorbed by the items to be stored in the closed container represents the major portion of the water vapor that has to be controlled in order to minimize or prevent deterioration. It is generally recognized that corrosion of metals does not occur at a significant rate in an atmosphere of 30 to 40 per cent relative humidity or below at 68 F. Although there is some evidence that the burning rate of explosives may be changed by prolonged exposure to low-humidity atmospheres, the Sponsor indicated that this possible effect of low humidity is not considered to be damaging. Therefore, it appeared that the main function of the desiccating capsule of interest should be to maintain the relative humidity of the atmosphere in a hermetically sealed storage container below approximately 40 per cent.

As described in our letter report dated July 12, 1955, that summarized the activity conducted under Research Order No. 23, a survey had been made of the information available on the properties and the factors affecting the efficiency of various desiccating agents. An analysis of the data obtained indicated that a chemically inert material would serve best in the intended application, as acidic and basic materials tend to promote corrosion rather than to prevent it.

Chemically inert desiccants may be divided into two classes: absorptive and adsorptive materials. Some materials, such as calcium sulfate, are absorptive because of their ability to tie up water vapor as water of hydration. They do not react with other vapors that may be present in the atmosphere, and usually have a relatively low capacity for water. Adsorptive materials, such as silica gel and activated alumina, are characterized by their ability to adsorb water physically. Some of these - silica

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gel, for example - will also remove vapors other than water vapor from the atmosphere. These materials generally have a high capacity for water and are relatively cheap.

For the above-indicated reasons, adsorptive materials were chosen for evaluation under Research Order No. 29. Silica gel was picked as the best of the class of adsorptive materials; of the various commercial silica gels, Protek-Sorb 121 was selected because it had the highest water-adsorption capacity, and also satisfied Specification MIL-D-3464. This grade of silica gel is commercially available; it is packaged in tough, resin-impregnated, water-vapor-permeable cellulose-paper bags of various suitable sizes, as well as in water-vapor-permeable perforated aluminum cans.

EXPERIMENTAL WORK AND RESULTS

Storage of Containers Loaded With Various Items

The humidity-measuring system used in this study consisted of humidity-sensing elements and an a-c indicator, which were obtained from the American Instrument Company, Incorporated, Silver Spring, Maryland. The sensing elements were of the special, wide-range, single-unit type. They covered a range of 15 to 55 per cent relative humidity with an accuracy of 3 per cent; above this range, the accuracy was not known. Correction curves made available by the manufacturer permitted the use of these elements at temperatures from 40 to 120 F. The humidity-measuring device was identified as American Instrument Company Electric Hygrometer Indicator No. 4-5170.

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A humidity-sensing element was installed in the lid of each of three of the Sponsor's rectangular-cross-sectioned (7 x 9 x 16-1/2 inch) stainless steel containers (approximately 0.6-cubic-foot internal capacity), and in the lid and in the bottom of a cylindrical container of the type developed under Research Order No. 16 (approximately 2.3-cubic-foot internal capacity). The installation procedure was as follows: A hole approximately 1 inch in diameter was drilled in the appropriate surface(s) of each container to permit insertion of and to provide clearance for the sensing element. Then, a neoprene-rubber gasket was slipped over the element so as to provide a seal between the element flange and the container surface. The element was inserted into the large hole, and four small holes were drilled through the gasket and the container surface in locations corresponding to those of the holes in the element flange. Machine screws and nuts were used to secure the sensing element to the container. During cycled storage, as described below, it became evident that small leaks were occurring around the gasket. To plug these, glyptal was painted liberally around the seal.

Each container was loaded with a variety of mutually agreed upon items* (Tables 1 and 2); enough packaged Protek-Sorb 121 silica gel was inserted in the large storage container and two of the small containers to provide approximately 1,000 grams of gel per cubic foot of container capacity. Eight-unit** bags of gel, as shown in Figure 1, were used; these were located at the bottom, middle, and top of the load in the large container and at the bottom and top of the loads in the small containers. The large container and

* Laboratory measurements were made on some of the electronic components, to provide a basis for comparison of selected characteristics before and after storage.

** Silica gel is generally marketed on the basis of commercial units, one commercial unit representing 24.8 grams of silica gel.

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Figure 1. Commercially Available Bags and Cans of Protek-Sorb 121 Silica Gel. The containers marked "Air-Dryer" are the outer cans in which the silica gel - containing cans are packed. Note that tape is used to aid in sealing the outer cans.

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one small container were loaded with silica gel under temperate ambient conditions of room temperature and about 50 per cent relative humidity. One small container was loaded with silica gel and one without silica gel under tropical conditions, i. e., 90 F and about 80 per cent relative humidity. All of the containers were then stored continuously for 8 hours of each day at 120 F followed by 16 hours at 40 F. Readings of the relative humidity of the enclosed atmospheres were taken at the end of each storage period at each temperature by means of the humidity indicator plugged into the outer end of the sensing element.

After 1 to 2 days of storage under these cycled conditions, the relative humidity in all of the containers dropped below 15 per cent, except in the container that was loaded without silica gel; the relative humidity in this container dropped to about 50 per cent. The containers were maintained on this storage cycle for an additional month, to determine any change; none occurred. The containers were removed from storage and reloaded under the same conditions as before, but with about 660 grams of silica gel per cubic foot. Within 2 days, the relative humidity in all of the containers was below 15 per cent except for that loaded without silica gel; the relative humidity in this container was above 55 per cent.

The use of 1,000 grams of silica gel per cubic foot reflected calculations made on the basis of several assumptions about the moisture absorptivity of the items packed in the containers. The above-described experiments indicated that 1,000 and 660 grams of silica gel per cubic foot reduced the relative humidity of the atmosphere in the packed containers too far. Therefore, it was decided that a series of short-term experiments with various

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amounts of silica gel should be conducted in order to determine the amount of gel needed to maintain the relative humidity in the packed containers at approximately 25 per cent during the storage cycling described above.

Subsequently, the containers were removed from storage* and opened, and the stored items were exposed to a tropical atmosphere to achieve equilibrium in regard to absorbed and adsorbed moisture. After equilibrium was reached, the containers were reloaded with the items, and with approximately 100 grams of silica gel per cubic foot, under the same conditions as previously. This was found to be an insufficient amount, as were approximately 150 and 200 grams per cubic foot. The silica gel charge was then increased to approximately 300 grams** per cubic foot, and storage tests were conducted for approximately six months.

Note: All items soaked in trop. environ. 1st. Then 2 containers loaded w/ first items. Other items soak in temperate environment before being loaded in containers.

During the six-month tests, relative-humidity data for all of the containers were obtained twice a day. At the end of the six-month storage period, all of the containers were opened and the stored items inspected. All of the items were examined visually. In some instances, simple, appropriate inspection tests were also conducted - such as igniting the wooden safety matches, fuzes, and rifle-shell powder; and checking the time delays provided by the time pencils. Laboratory measurements were made on several electronic components, to permit a comparison of selected characteristics before and after storage. The results obtained are described below.

Relative Humidity Versus Storage Time Data

Plots of relative humidity versus storage time for all of the containers are presented in Figures 2, 3, and 4.

* The container that was loaded under tropical conditions without silica gel was not removed from storage.

** This represents about 12 commercial (Davison Chemical Company) units of silica gel.

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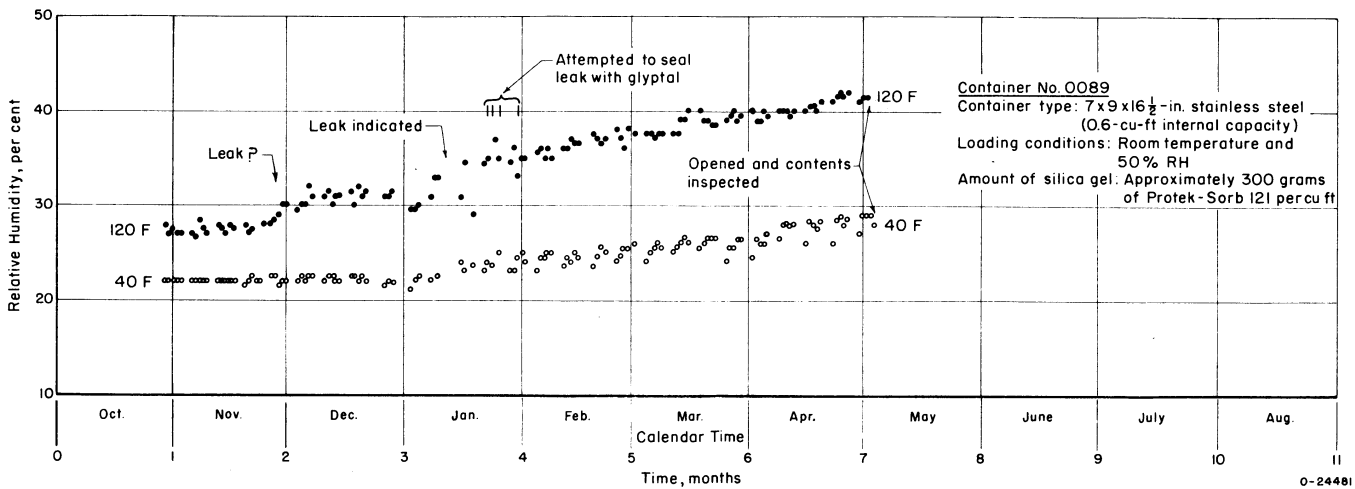


FIGURE 2. DATA ON RELATIVE HUMIDITY VERSUS STORAGE TIME FOR SMALL CONTAINER LOADED UNDER TEMPERATE CONDITIONS, WITH SILICA GEL

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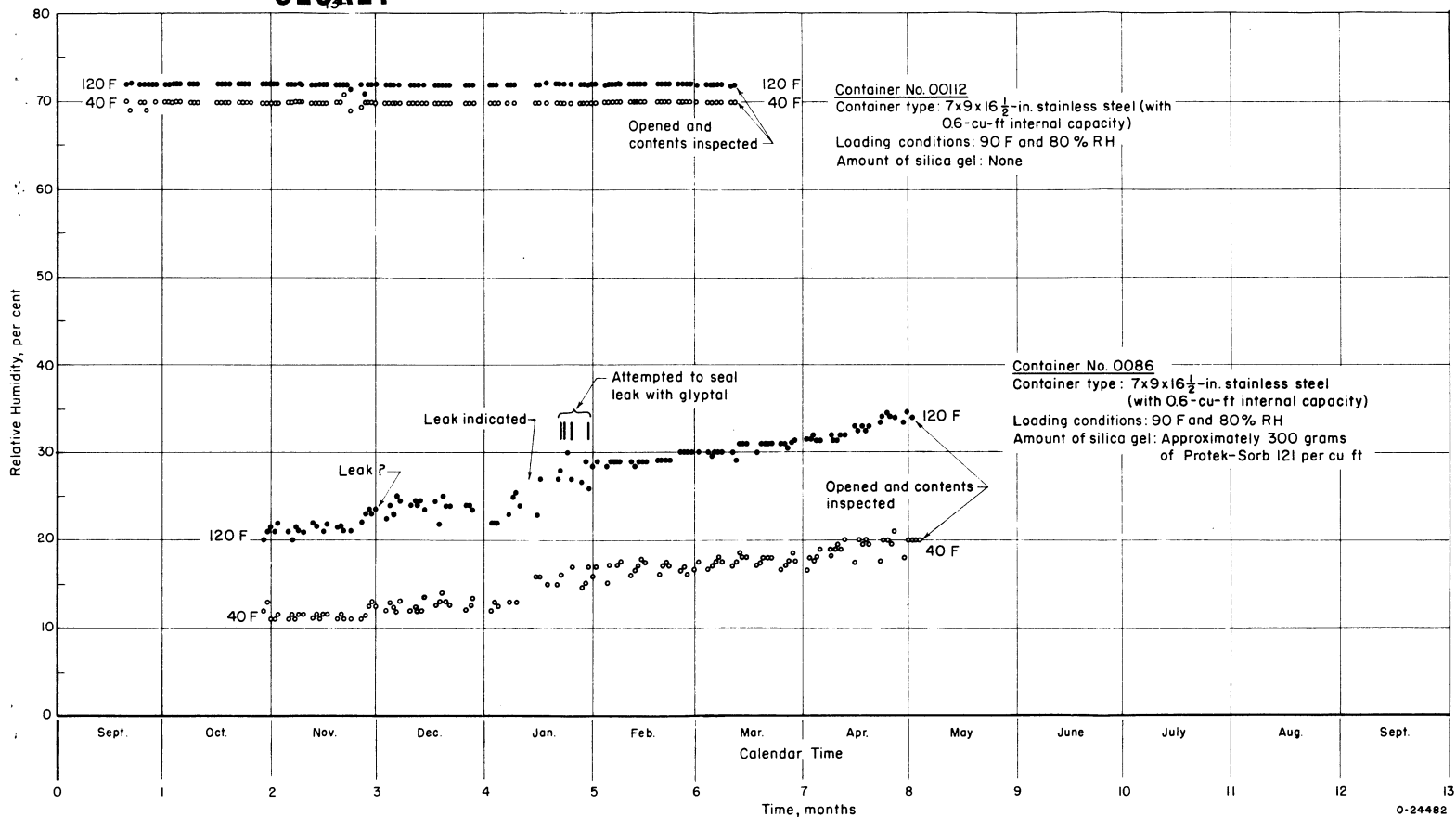
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FIGURE 3. DATA ON RELATIVE HUMIDITY VERSUS STORAGE TIME FOR TWO SMALL CONTAINERS LOADED UNDER SIMULATED-TROPICAL CONDITIONS, WITH AND WITHOUT SILICA GEL

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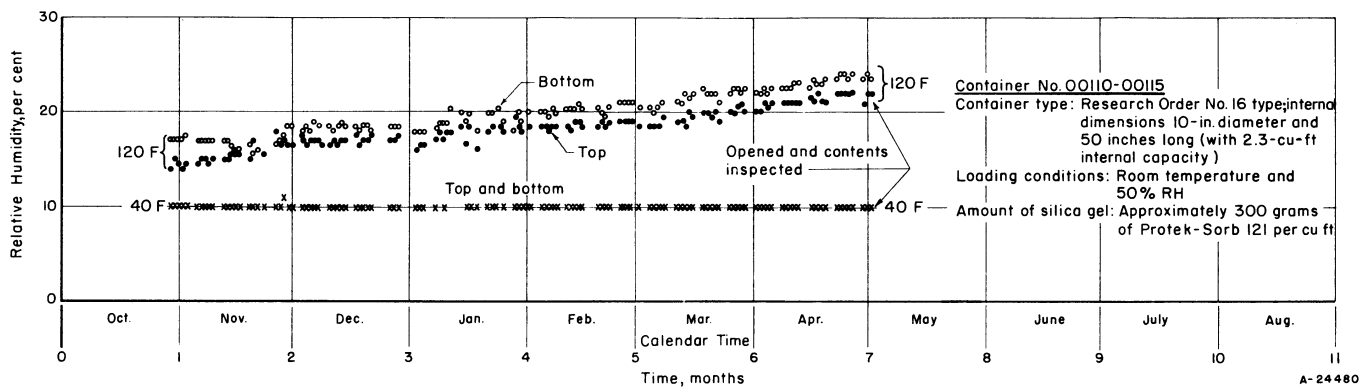


FIGURE 4. DATA ON RELATIVE HUMIDITY VERSUS STORAGE TIME FOR LARGE CONTAINER LOADED UNDER TEMPERATE CONDITIONS, WITH SILICA GEL

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During the six-month period of cycled storage, approximately 300 grams of silica gel per cubic foot maintained the relative humidity of the atmosphere inside each of the three silica gel - loaded containers below 35 per cent, as compared to a relative humidity of about 70 to 72 per cent for the container without silica gel. However, the data generally indicated a gradual increase in the relative humidity for the three containers with increasing storage time; on the basis of the apparent rate of increase, which was relatively constant, it appeared that this "concentration" of silica gel would not control the relative humidity over long periods of time, such as two years, to the extent desired. This apparent increase in the relative humidity is attributed to three factors: (1) intermittent leakage around the seal between the sensing element and the container, (2) the strong tendency for water vapor to permeate a watertight seal between a relatively humid atmosphere and a relatively dry atmosphere, and (3) the possibility of changes in the calibration of the sensing elements in applications of this type over relatively long periods of service, such as six months.

With regard to leakage, readings were taken on each container twice a day, and, in the course of making the electrical connection between the humidity indicator and the sensing element, generally some strain was imposed on the sensing element - container seal. In certain instances, as indicated on the curves in Figure 2 and on the lower set of curves in Figure 3, the occurrence of leaks was noted or suspected, and attempts were made to plug them by applying glyptal. Also, the once-a-day thermal cycling to which the containers were subjected had a straining effect on the glyptal used to plug the pinhole-type leaks, and possibly also on the seal in general.

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A major portion of the apparent rate of increase in relative humidity indicated by the curves in Figure 2 and the lower part of Figure 3 is attributed to leakage occurring in this manner. However, the curves for the large container in Figure 4 show less of an apparent rate of relative-humidity increase than do those in Figure 2 and in the lower part of Figure 3 for the small containers. It is believed that the above-described type of leakage did not occur significantly on the large container; as a result of its greater over-all ruggedness, this container had a lower "normal leak rate"* than did the small container, and also its sensing-element seals were probably less sensitive to the type of handling involved in making an electrical connection with the humidity indicator.

The second major factor contributing to the apparent increase in humidity was probably water-vapor permeation of the storage-container water-tight seal. During a large portion of the cycled-storage period, the sensing element - container seal may have been tight enough to exclude liquid water. However, there is a strong tendency for water vapor to migrate to a relatively dry atmosphere under conditions such as those that prevailed in these experiments. Substantially all of the increase in relative humidity for the large container and a significant proportion of that for the small containers with silica gel (Figure 2 and lower part of Figure 3) are attributed to this factor.)

Handwritten note on right margin:
The humidity indicator is not airtight. It is possible that some of the humidity increase is due to leakage from the indicator itself.

The third contributing factor might have been changes in the calibration of the sensing elements. This is not considered to be of major importance. However, at the invitation of the manufacturer of the sensing elements, the units will be returned for re-calibration, gratis. The information

* It is generally recognized that even the so-called "tight" systems, whether water or vacuum tight, have finite, although very low, leak rates.

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ultimately obtained in this regard will be transmitted to the Sponsor as a supplement to this report.

The relative humidity in the small container loaded without silica gel remained constant in the range of about 70 to 72 per cent (see top portion of Figure 3), as was expected. The absence of any increase in relative humidity is attributed to the relative insensitivity of the sensing elements at high humidities.

Under actual service conditions, a closed storage container of either of the types used in this study would have only one, watertight seal. Consequently, leakage of the type encountered around the sensing element - container seal would not be expected to occur. The increase in relative humidity attributed to water-vapor permeation of the closure seal is probably unavoidable in the course of long-term storage. However, the rate of increase involved in the large container (Figure 4) amounted to approximately 1 per cent per month at 120 F. This might be most practically compensated for by using extra silica gel in sufficient amounts to maintain the relative humidity in the range of interest for the desired storage period. On the basis of calculations, it is believed that an additional 150 grams of Protek-Sorb 121 per cubic foot would adsorb the amount of water vapor corresponding to a relative-humidity increase of approximately 1 per cent per month at 120 F, and thus would maintain in these types of containers a relative humidity of approximately 20 per cent at 120 F for about 24 months.

It is recommended that long-term storage tests be conducted to check out the effectiveness of approximately 450 grams of Protek-Sorb 121 per cubic foot of internal container capacity in maintaining the relative humidity of

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the atmosphere enclosed in storage containers below the level that leads to corrosion of and damage to items stored for long periods.

Results of Inspection of Stored Items

The results obtained from the inspection of the stored items are presented in Tables 1 and 2.

The items stored in the one large and two small containers that were loaded with silica gel fared approximately the same, respectively. This was to be expected because they were all subjected to approximately the same conditions of relative humidity and temperature during the six months of cycled storage.

The Grade C greaseproof paper in all of the containers apparently melted during the storage periods at 120 F. The pressure-sensitive adhesives on some of the items such as the Scotch cellophane tape and medical adhesive tape that were stored with silica gel present generally showed slightly decreased tackiness. One binocular case, from a container with silica gel, exhibited a small amount of mold* on the inside surface.

All of the B and C batteries showed signs of deterioration, and three of each type, from containers with and without silica gel, were dead. The separate steel flashlight batteries (not inside the flashlight case) that were in the containers with silica gel were all in good condition, with no loss in voltage; those in the container without silica gel were rusted and dead. The steel flashlight batteries that were loaded inside flashlight cases during storage in the containers with silica gel all had deteriorated

* The mold was of genus Gliocladium, which is a common penicillium that grows on generally all types of materials throughout the world.

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TABLE 1. RESULTS OF QUALITATIVE INSPECTION OF ITEMS STORED
IN CONTAINERS FOR SIX MONTHS

Item	Condition of Item in Indicated Container			
	Large, Loaded "Dry", With Gel (1)	Small, Loaded "Dry", With Gel (1)	Small, Loaded "Wet", With Gel (2)	Small, Loaded "Wet", No Gel (2)
Putty, in wax paper	OK	OK	OK	OK
Dummy blasting cord	OK	OK	OK	OK
Safety fuse	OK	OK	OK	OK
Dummy thermite well	OK	OK	OK	OK
Wooden safety matches (small), in box	OK	OK	OK	OK
Fuzees, in box	OK	OK	OK	OK
Binoculars and leather case	Mold in case interior	OK	OK	OK
Document paper with ink writing	OK	OK	OK	OK
Electrical wall switch	OK	OK	OK	OK
Time pencils, in taped case	OK	OK	OK	Case and brass parts corroded slightly, operative
Electronic components		All OK appearancewise; see Table 2		
Rifle-shell powder, in plastic capsule	OK	OK	OK	OK
Pistol, in protective paper	OK	OK	OK	Bore rusted, inoperative
Pull-type firing device	Appeared OK	Appeared OK	Appeared OK	Appeared OK
Iron wire, on wooden spool	OK	OK	OK	Rust spots

TABLE 1. (Continued)

Item	Condition of Item in Indicated Container			
	Large, Loaded "Dry", With Gel (1)	Small, Loaded "Dry", With Gel (1)	Small, Loaded "Wet", With Gel (2)	Small, Loaded "Wet", No Gel (2)
B battery	Dead	Distorted, voltage low	Leaked, dead	Asphalt bad, dead
C battery	Dead	Ditto	Ditto	Ditto
Separate steel-cased flashlight batteries	OK	OK	OK	Rusted, dead
Flashlight case (loaded with batteries)	OK	OK	OK	Corroded, inoperative
Two batteries (loaded in flash- light)	One dead	One dead	Voltage low	Rusty, dead
Blued crescent wrench	OK	OK	OK	Several rust spots
Blued wire cutter	OK	OK	OK	Ditto
Blued pliers	OK	OK	OK	Ditto
Blued metal shears	OK	-	-	-
Hand saw	OK	-	-	-
Claw hammer	OK	OK	OK	OK
Screw driver (plastic handle)	OK	OK	OK	Several rust spots
Scotch cellophane tape	Decreased adhesion	Decreased adhesion	Decreased adhesion	OK
Scotch electrical tape	OK	OK	OK	OK
Medical adhesive tape	Decreased adhesion	Decreased adhesion	OK	OK
Rubberized fabric	OK	OK	OK	OK
Silk cloth	OK	OK	OK	OK
Nylon cloth	OK	OK	OK	OK
Cotton cloth	OK	OK	OK	OK

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TABLE 1. (Continued)

Item	Condition of Item in Indicated Container			
	Large, Loaded "Dry", With Gel (1)	Small, Loaded "Dry", With Gel (1)	Small, Loaded "Wet", With Gel (2)	Small, Loaded "Wet", No Gel (2)
Polyethylene sheet	OK	OK	OK	OK
Polyvinylchloride sheet	OK	OK	OK	Signs of de- terioration
Plexiglas sheet	OK	OK	OK	OK
Polystyrene foam	OK	OK	OK	OK
Grade C greaseproof paper	Poor	Poor	Poor	Poor
Wood	OK	OK	OK	OK
Enameled carbon-steel sheet	OK	OK	OK	OK
Unpainted carbon-steel sheet	OK	OK	OK	Several rust spots
Magnesium sheet	OK	OK	OK	OK
Aluminum sheet	OK	OK	OK	OK
Copper sheet	OK	OK	OK	OK
Brass sheet	OK	OK	OK	Some corrosion

- (1) Loaded at room temperature and about 50 per cent relative humidity, with about 300 grams of Protek-Sorb 121 silica gel per cubic foot of container capacity.
- (2) Loaded at 90 F and about 80 per cent relative humidity, with about 300 grams of Protek-Sorb 121 silica gel per cubic foot of container capacity.

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TABLE 2. DATA ON ELECTRONIC COMPONENTS BEFORE
AND AFTER SIX-MONTH CYCLED STORAGE
IN VARIOUS CONTAINERS

Component	Initial Reading	Reading After Storage	Storage Variance, per cent	Tolerance, per cent
<u>Large Container, Loaded "Dry"(1), With Gel</u>				
Molded silver mica fixed capacitor, Type CM 25C 102M, 1,000 $\mu\mu\text{f}$, 500 volts	1,020.0 $\mu\mu\text{f}$	1,019.5 $\mu\mu\text{f}$	-0.05	20
Glass dielectric fixed capacitor, Type Cy 20 C102J, 1,000 $\mu\mu\text{f}$, 500 volts	899.5 $\mu\mu\text{f}$	900.3 $\mu\mu\text{f}$	+0.1	5
Plastic molded R-F choke, Type LT8K007, 15 microhenries	16.2 μh	17.0 μh	+4.9	10
Molded borocarbon fixed resistor, 1/2 watt, 0.5 megohm	500,800 Ω	502,300 Ω	+0.3	1
Coated borocarbon fixed resistor, Type RC-25, 1/2 watt, 0.5 megohm	499,100 Ω	500,260 Ω	+0.2	1
Solid-composition fixed resistor, Type RC-20, BF 504, 1/2 watt, 0.5 megohm	511,000 Ω	508,360 Ω	-0.5	5
<u>Small Container, Loaded "Dry"(1), With Gel</u>				
Molded silver mica fixed capacitor, Type CM 25C 102M, 1,000 $\mu\mu\text{f}$, 500 volts	973.5 $\mu\mu\text{f}$	972.6 $\mu\mu\text{f}$	-0.1	20

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TABLE 2. (Continued)

Component	Initial Reading	Reading After Storage	Storage Variance, per cent	Tolerance, per cent
Glass dielectric fixed capacitor, Type Cy 20 C102J, 1,000 μ f, 500 volts	849.4 μ f	849.4 μ f	0.0	5
Plastic molded R-F choke, Type LT8K007, 15 microhenries	17.0 μ h	17.0 μ h	0.0	10
Molded borocarbon fixed resistor, 1/2 watt, 0.5 megohm	498,800 Ω	500,460 Ω	+0.3	1
Coated borocarbon fixed resistor, Type RC-25, 1/2 watt, 0.5 megohm	500,000 Ω	501,480 Ω	+0.3	1
Solid-composition fixed resistor, Type RC-20, BF 504, 1/2 watt, 0.5 megohm	509,500 Ω	513,600 Ω	+0.8	5
<u>Small Container, Loaded "Wet"(2), With Gel</u>				
Molded silver mica fixed capacitor, Type CM 25C 102M, 1,000 μ f, 500 volts	965.3 μ f	964.7 μ f	-0.1	20
Glass dielectric fixed capacitor, Type Cy 20 C102J, 1,000 μ f, 500 volts	893.1 μ f	893.8 μ f	+0.1	5
Plastic molded R-F choke, Type LT8K007, 15 microhenries	16.3 μ h	16.7 μ h	+2.5	10

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TABLE 2. (Continued)

Component	Initial Reading	Reading After Storage	Storage Variance, per cent	Tolerance, per cent
Molded borocarbon resistor, 1/2 watt, 0.5 megohm	501,400 Ω	503,400 Ω	+0.4	1
Coated borocarbon fixed resistor, Type RC-25, 1/2 watt, 0.5 megohm	500,500 Ω	501,960 Ω	+0.3	1
Solid-composition fixed resistor, Type RC-20, BF 504, 1/2 watt, 0.5 megohm	513,100 Ω	516,290 Ω	+0.6	5
<u>Small Container, Loaded "Wet"(2), No Gel</u>				
Molded silver mica fixed capacitor, Type CM 25C 102M, 1,000 $\mu\mu\text{f}$, 500 volts	974.5 $\mu\mu\text{f}$	974.0 $\mu\mu\text{f}$	-0.1	20
Glass dielectric fixed capacitor, Type Cy 20 C102J, 1,000 $\mu\mu\text{f}$, 500 volts	885.0 $\mu\mu\text{f}$	885.0 $\mu\mu\text{f}$	0.0	5
Plastic molded R-F choke, Type LT8K007, 15 microhenries	16.6 μh	16.9 μh	+1.8	10
Molded borocarbon resistor, 1/2 watt, 0.5 megohm	499,400 Ω	507,840 Ω	+1.7	1

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TABLE 2. (Continued)

Component	Initial Reading	Reading After Storage	Storage Variance, per cent	Tolerance, per cent
Coated borocarbon fixed resistor, Type RC-25, 1/2 watt, 0.5 megohm	499,500 Ω	502,500 Ω	+0.6	1
Solid-composition fixed resistor, Type RC-20, BF 504, 1/2 watt, 0.5 megohm	515,100 Ω	541,800 Ω	+5.2	5

Note: In the one large and two small containers, approximately 300 grams of Protek-Sorb 121 was used per cubic foot of container capacity.

- (1) Loaded at room temperature and about 50 per cent relative humidity.
- (2) Loaded at 90 F and about 80 per cent relative humidity.

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voltage-wise, but not in appearance, and two of these batteries in different flashlight cases were completely dead; the corresponding flashlight cases were in good condition. Without silica gel present in the container, both of the batteries stored in the flashlight case were rusty and dead; the corresponding flashlight case was corroded and inoperative.

The pistol stored without silica gel showed a rusty bore and was inoperative. The pistols stored with silica gel were in good condition.

Some of the materials showed slight corrosion or signs of slight deterioration when silica gel was not present, but the damage was not significant.

Two resistors stored under simulated-tropical conditions without silica gel showed gains beyond the tolerance limits; however, this is not unusual for units of this type, even under normal storage conditions. The electronic components, as a whole, were not affected by storage under any of the conditions that prevailed in this study.

Storage of Containers Loaded With Cushioning Material

A few experiments were conducted on the storage of four cushioning materials with silica gel present. The Sponsor's rectangular-cross-sectioned stainless steel containers and the humidity-measuring system which were described above were used in these experiments also. The four cushioning materials investigated included Kimpak and Hair-lok, furnished by the Sponsor, and two grades of Tufflex, normal and non water absorbing, that were obtained from the Wood Conversion Company, St. Paul, Minnesota, at the suggestion of the Sponsor.

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Approximately one-half cubic foot of each of the cushioning materials was packed into the 0.6-cubic-foot containers individually under simulated-tropical conditions, i. e., 90 F and about 80 per cent relative humidity. As in the case of the previously described experiments, approximately 300 grams of Protek-Sorb 121 silica gel per cubic foot of container capacity was also loaded into each container. All of the containers were then subjected to the above-described cycled storage for approximately one month; relative-humidity readings were taken at the end of each storage period at each temperature.

Within 1 to 2 days of storage, the atmospheres in the containers loaded with Kimpak and Hair-lok dropped to 40 per cent relative humidity at 40 F, and 50 per cent relative humidity at 120 F. Within the same period of time, the containers packed with the two grades of Tufflex yielded relative-humidity values above 70 per cent. For all of the containers, the above-indicated relative-humidity values persisted throughout the one-month duration of the experiments.

Apparently, when container loading occurs under tropical conditions, these two grades of Tufflex absorb considerable water vapor. For this reason, it appears that they would not be so good as Kimpak and Hair-lok for use in containers under service conditions involving tropical loading and long-term closed storage.

Storage of Commercial Silica Gel Packages
Under Simulated-Tropical Conditions

A cursory investigation was conducted to obtain some indication of the ability of commercial silica gel packages to resist moisture permeation during shelf storage. In considering the potential application of silica

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gel "capsules" in long-term closed storage, the Sponsor had indicated that the use of a metal can rather than a fabric or paper bag to "house" the silica gel would be advantageous. In the actual loading of a storage container, it was anticipated that the silica gel capsules would be in contact with the materiel being packed; after the silica gel had picked up water vapor during storage, it would be moist, and consequently, if it was held in a bag, the bag would ultimately become moist. This condition might lead to localized corrosion of the material contacted by this sort of silica gel package. On the other hand, if the silica gel were packaged in a metal can, the probability of this type of localized corrosion occurring would be extremely slight. Therefore, silica gel packaged in metal cans was used in the experiments described here.

In this category of silica gel packages, the material is commercially available in water-vapor-permeable, perforated aluminum cans of two shapes. One is circular in cross section and is approximately 2-3/4 inches in diameter by 5/8 inch in thickness; the other is rectangular cross sectioned with round corners and measures approximately 4 by 2 by 1/2 inch over-all (see Figure 1). In both instances, the silica gel cans are packed for shipment and storage in outer friction-top metal containers that conform to the shape of the respective silica gel cans; plastic sealing tape is wrapped around the sides of the outer containers to aid in maintaining the seal. Both the circular- and rectangular-cross-sectioned packages shown in Figure 1 contain one commercial unit (24.8 grams) of silica gel.

In this investigation, four silica gel packages of each shape including the outer cans and sealing tape were selected at random for storage

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for one month under the most adverse conditions anticipated in service, i. e., under simulated-tropical conditions (at 90 F and about 80 per cent relative humidity). The sealing tape was removed from two packages of each shape. Then, each of the eight packages was weighed and stored in our Tropical Room. At the end of one month, the eight packages were re-weighed, to determine the amount of water vapor that had been picked up by the package. The weight data are presented in Table 3.

The circular-cross-sectioned outer cans provided slightly better protection against water-vapor permeation than did the rectangular-cross-sectioned outer cans, with or without the sealing tape, respectively. This was expected, because the circular shape is more conducive to a better friction-type seal. The sealing tape aided significantly in sealing the outer cans of either shape. The circular-cross-sectioned outer can with the sealing tape intact showed substantially no water vapor pickup. Consequently, it is believed that this packaging arrangement would satisfy the requirements of the shelf-storage and handling procedures necessarily involved in the ultimate application of silica gel capsules under service conditions.

DISCUSSION OF APPLICATION AND FUTURE
LOGISTICS CONSIDERATIONS

During the course of this research program, consideration was given to the packaging of the silica gel for use in this application. Several possible methods of packaging the silica gel were reviewed with the Sponsor; these were directed toward providing the optimum in protection against moisture permeation during shelf storage, in ease and versatility of

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TABLE 3. DATA ON MOISTURE PICKUP BY CIRCULAR-
AND RECTANGULAR-CROSS-SECTIONED SILICA
GEL PACKAGES UNDER SIMULATED-TROPICAL
CONDITIONS* FOR ONE MONTH

Type of Package**	Weight, grams		Gain
	Before Storage	After Storage	
<u>Circular cross section</u>			
Sealing tape intact	80.0	80.0	0.0
Ditto	82.5	82.6	0.1
Sealing tape removed	83.1	88.2	5.1
Ditto	83.4	90.2	6.8
<u>Rectangular cross section</u>			
Sealing tape intact	89.0	89.4	0.4
Ditto	90.0	90.9	0.9
Sealing tape removed	89.0	99.0	10.0
Ditto	90.0	100.0	10.0

Note: Figure 1 illustrates these two types of packages.

* At 90 F and about 80 per cent relative humidity.

** Each package included the silica gel held in a perforated aluminum container, plus the outer shipping and storage metal can; as indicated in this column, some of the tests were conducted with the plastic sealing tape intact, and some with the sealing tape removed.

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application, and in over-all effectiveness in maintaining control of the relative humidity. In the final analysis, it appears prudent first to decide on the amount of silica gel to be contained in a "standard" package, such a package to be utilized in multiples, depending on the internal capacity of the storage containers that the Sponsor anticipates using. It appears, on the basis of the laboratory data and calculations, that 80 to 100 grams of silica gel per package would represent a useful "standard" size. Of the commercially used shapes of silica gel packets, the circular cross section is considered to be the best, particularly from the standpoint of the outer storage and shipping can. The use of a circular-cross-sectioned outer can with plastic sealing tape wrapped around the periphery is expected to minimize moisture permeation of the "standard" package during shelf storage.

In this connection, to obtain some order-of-magnitude indications of the number of "standard" silica gel packages needed per storage container and the volume occupied by those packages, calculations were made using a range of pertinent parameters. The calculations were based on 80 to 100 grams of Protek-Sorb 121 per "standard" circular-cross-sectioned package; on a required "concentration" of 300 to 450 grams of Protek-Sorb 121 per cubic foot of container internal capacity; and on the use of the two types of containers (approximately 0.6 and 2.3-cubic-foot internal capacity) in which the previously described experiments were conducted. The volume that would be occupied by the 80 to 100 grams of silica gel was taken to be in direct relation to that for 24.8 grams of silica gel. The results of the calculations are summarized as follows:

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<u>Factor of Interest</u>	<u>Small Container (1,040 cu. in.)</u>	<u>Large Container (3,930 cu. in.)</u>
No. of standard 80 to 100-gram packages needed	2 to 4	7 to 13
Volume occupied by above number of standard packages	30 to 48 cu. in.	105 to 165 cu. in.
Volume, per cent, of container occupied by above number of standard packages	2.9 to 4.6 per cent	2.7 to 4.2 per cent

It is contemplated that, for service applications, the required number of standard packages could be loaded at the top and bottom or interspersed throughout the load.

After at least tentative decisions are made by the Sponsor with regard to the weight of silica gel and the types of cans to be used for the standard package, it is suggested that the Sponsor negotiate with a manufacturer of silica gel, so as to utilize his specialized background of experience in the over-all design of the "standard" silica gel package and also, if desired, to obtain some production lots of the item. To explore the interest of a manufacturer in producing packages of silica gel that are different from currently available packages and to obtain an indication of the costs involved, we contacted the Davison Chemical Company, Division of W. R. Grace and Co., Baltimore, Maryland. This organization indicated a definite interest in producing special silica gel packages and quoted estimated prices for providing 80 grams of silica gel packaged in metal cans, in two production quantities; their letter of quotation is appended for the record.

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RECOMMENDATIONS

The laboratory investigation described in this report has demonstrated the effectiveness of Protek-Sorb 121 silica gel in maintaining the desired relative-humidity control in the application of interest for relatively long periods of storage. It appears prudent, in this instance also, to check out the laboratory results by performing long-term field storage tests. In such tests, appropriate equipment and materials packaged in accord with the "standardized" procedures should be used, and the effectiveness of Protek-Sorb 121 silica gel in two different amounts - approximately 300 and 450 grams per cubic foot of internal container capacity - should be evaluated. The loading of the containers should be done under the most rigorous conditions, for example, under conditions involving high relative humidity. In view of the relatively low cost of the silica gel, it is believed that the Sponsor might consider packing into all storage containers the amount of silica gel needed to maintain control under the worst conditions anticipated.

With regard to finalizing the over-all design of a standard silica gel package (including the outer can), it is recommended that the Sponsor discuss the item of interest with a manufacturer of silica gel so as to utilize his vast specialized experience in this connection. Also, the groundwork can be accomplished for obtaining production lots of the item, if desired.

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APPENDIX

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DAVISON CHEMICAL COMPANY

DIVISION OF W. R. GRACE & CO.

BALTIMORE 3 MARYLAND

INDUSTRIAL CHEMICALS DEPARTMENT

REPLY TO:

REGIONAL OFFICE
918 ATLAS BUILDING
COLUMBUS 15, OHIO

April 26, 1957

We wish to quote the following prices on the X-1220 Air Dryers Containing 80 grams of gel in each dryer.

25000 per year
\$1.02 per dryer

50000 per year
\$.836 per dryer

Dryers to be packed six per carton and will have outer lithographed container.

25000 per year
\$.515 per dryer

50000 per year
\$.425 per dryer

Dryers packed 78 per 5 gal can. Dryers will not have outer lithographed container.

The above prices are F.O.B. our plant, Curtis Bay, Baltimore, Maryland.

If your sponsorer uses the dryers on a continuing process over a longer period of time we may be able to revise the above prices.

Very truly yours,
Davison Chemical Company
Division of W. R. Grace & Co.
Columbus, Ohio

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